IS 7976 (1976): Phorate, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

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Indian Standard

SPECIFICATION FOR PHORATE, TECHNICAL

(Fourth Reprint JUNE 1999)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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SPECIFICATION FOR PHORATE, TECHNICAL

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AMENDMENT NO. 1 FEBRUARY 1985

TO

IS : 7976 - 1976 SPECIFICATION FOR PHORATE, TECHNICAL

(*Page* 6, *Table* 1):

a) *Col 5* — Substitute ' IS : 6940-1982* ' *for* ' IS · 6940-1973 *'.

b) *Foot-note with* ' * ' *mark* — Substitute the following for the existing foot-note:

' *Methods of test for pesticides and their formulations (*first revision*).'

(*Page* 6, *clause* **3.1**) — Substitute the following for the existing clause:

'**3.1 Packing** — The material shall be packed as per requirement given in IS : 8190 (Part II)-1980*.'

(*Page* 6) — Add the following foot-note·

' *Requirements for packing of pesticides. Part II Liquid pesticides (*first revision*).'

[*Page* 7, *clause* **3.2**(g)] — Delete.

(*Page* 7, *clause* **3.2.1**) — Delete.

(*Page* 7, *clause* **4.1** *and note*) — Substitute the following for the existing matter:

'**4.1** Representative samples of the material shall be drawn according to IS : 10946-1984*'.

(*Page* 7, *clause* **5.2**, *line* 2) — Substitute ' IS : 1070-1977† ' *for* ' IS : 1070-1960† '.

(*Page* 7, *foot-notes with* ' * ' *and* ' † ' *marks*) — Substitute the following for the existing foot-notes:

' *Methods for sampling of technical grade pesticides.
†Specification for water for general laboratory use (*second revision*).'

(*Page* 10, *clause* **A-2.3.1**, *equation*) — Substitute the following for the existing equation:

'**A-2.3.1** Phorate content, percent by mass

$$= \left[\frac{6{\cdot}510 \times \{(V_1 F_1) - (V_2 F_2)\}}{M_1}\right] - \left[\frac{1{\cdot}302 \times \{(V_1 F_1) - (V_2 F_2)\}}{M_2}\right]$$

(AFCDC 6)

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AMENDMENT NO. 2 OCTOBER 2010
TO
IS 7976 : 1976 SPECIFICATION FOR PHORATE, TECHNICAL

(Page 6, *clause* 2.2, *Table I, SI No.* 1, *col* 4) — Substitute 'Clause 3.3.1 of IS 9359 : 1995†' *for* 'A'.

(Page 6, *clause* 2.2, *Table* 1) — Add the following footnote at the end:

'**†Pesticide — Phorate eucapsulated — Specification** *(first revision).*'

(FAD 1)

Reprography Unit, BIS, New Delhi, India

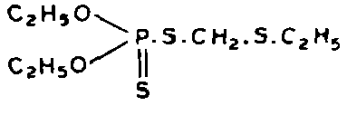
Indian Standard
SPECIFICATION FOR PHORATE, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 25 February 1976, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Phorate is an organophosphorus soil and systemic insecticide used for protection against sucking and chewing insects, mites and certain neuratodoes infesting various crops.

0.3 Phorate is the accepted common name by the International Organization for Standardization (ISO) for O,O-diethyl S-[(ethylthio)-methyl] phosphorodithioate. Its empirical and structural formulae and molecular weight is as given below:

Empirical Formula	*Structural Formula*	*Molecular Weight*
$C_7H_{17}O_3PS_3$		260·4

0.4 In the preparation of this standard due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for phorate, technical.

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be yellow to amber coloured liquid, free from foreign matter and added modifying agents.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR PHORATE, TECHNICAL

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Appendix	Cl No. of IS : 6940-1973*
(1)	(2)	(3)	(4)	(5)
i)	Phorate content, percent by mass, *Min*	90	A	—
ii)	Material insoluble in acetone, percent by mass, *Max*	0·5	—	9
iii)	Moisture content, percent by mass, *Max*	0·5	—	4.1
iv)	Acidity (as H_2SO_4), percent by mass, *Max*	0·5	—	11.3.2
v)	Specific gravity at 25°C, *Min*	1·00	—	5

*Methods of tests for pesticides and their formulations.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in clean and dry mild steel containers which are suitably lacquered

3.2 Marking — The containers shall be securely closed and shall bear legibly and indelibly the following information in addition to the information as is necessary under the Insecticides Act and Rules:

a) Name of the material,

b) Name of the manufacturer,

c) Date of manufacture,

d) Batch number,

e) Phorate content,

f) Net mass of contents, and

g) The minimum cautionary notice worded as under:

HANDLE WITH CARE. KEEP OUT OF REACH OF CHILDREN AND WELL AWAY FROM FOODSTUFFS, ANIMAL FEEDS, AND THEIR CONTAINERS. AVOID SKIN CONTACT. WHILE HANDLING WEAR PROTECTIVE GLOVES AND CLEAN PROTECTIVE CLOTHING. IN CASE OF POISONING CALL PHYSICIAN SPECIFIC ANTIDOTES — ATROPINE AND PRALIDOXIME

3.2.1 In addition to the above, the container shall also be marked with the symbol for danger of poisoning as specified in IS : 1260 (Part I)-1973*.

3.2.2 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn according to the method prescribed in 'Indian Standard methods for sampling of pesticides and their formulations (*under preparation*)'.

NOTE — Till such time this standard is published, the samples shall be drawn as agreed to between the parties concerned

5. TESTS

5.1 Tests shall be carried out by the methods prescribed under col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS . 1070-1960†) shall be employed in tests

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis

*Pictorial marking for handling and labelling of goods. Part I Dangerous goods (*first revision*).

†Specification for water, distilled quality (*revised*)

APPENDIX A

[*Table* 1, *Item* (i)]

DETERMINATION OF PHORATE CONTENT

A-0. PRINCIPLE

A-0.1 Phorate is hydrolysed in slightly aqueous acid in the presence of standard silver nitrate solution. The hydrolysis products consume two moles of silver The excess silver is titrated with standard ammonium thiocyanate solution. A correction for the amount of standard silver nitrate solution consumed by impurities present before hydrolysis is applied. Total silver consumed gives a measure of the active ingredient content.

A-1. REAGENTS

A-1.1 Acetone

A-1.2 Nitric Acid — concentrated (*see* IS : 264-1968*).

A-1.3 Standard Silver Nitrate Solution — 0 1 N, prepared by dissolving 17·5 g silver nitrate in 1 000 ml of water, and standardized.

A-1.3.1 *Standardization of Standard Silver Nitrate Solution* — Pipette or measure accurately, about 40 ml of silver nitrate solution, and add slowly, with continuous stirring, dilute hydrochloric acid until precipitation of the silver is complete. Boil the mixture cautiously for about 5 minutes; then, allow it to stand in the dark until the precipitate has settled and the supernatant liquid has become clear. Transfer the precipitate completely to a tared filtering crucible, and wash it with small portions of water slightly acidified with nitric acid Dry the precipitate at 110°C to constant mass. From the mass of the silver chloride obtained, calculate the normality of the silver nitrate solution

A-1.3.1.1 *Calculations*

a) Normality of standard silver nitrate solution, $N_1 = \frac{m \times 6{\cdot}98}{v}$

where

m = mass in g of silver chloride, and

v = volume in ml of silver nitrate solution consumed.

b) Factor of normality, $F_1 = \frac{N_1}{0\,1}$

*Specification of nitric acid (*first revision*)

A-1.4 Nitrobenzene

A-1.5 Ferric Nitrate Solution — 10 percent.

A-1.6 Standard Ammonium Thiocyanate Solution — 0·1 N, prepared by dissolving 8 g of ammonium thiocyanate in 1 000 ml of water, and standardized.

A-1.6.1 *Standardization of Ammonium Thiocyanate Solution* — Measure accurately about 30 ml of standard silver nitrate solution (**A 1.3**) into a glass-stoppered flask. Dilute with 50 ml of water, add 2 ml of nitric acid and 2 ml of ferric ammonium sulphate indicator and titrate with ammonium thiocyanate solution to the first appearance of a reddish-brown colour.

A-1.6.1.1 *Calculations*

a) Normality of ammonium thiocyanate solution, $N_2 = \frac{vn}{V}$

where

v = volume in ml of silver nitrate solution,

n = normality of standard silver nitrate solution, and

V = volume in ml of ammonium thiocyanate solution consumed.

b) Factor of normality, $F_2 = \frac{N_2}{0 \cdot 1}$

A-1.7 Benzene

A-1.8 Standard Potassium Hydroxide Solution — 0·1 N, aqueous in 10 percent potassium nitrate.

A-1.9 Congo Red Test Paper

A-2. PROCEDURE

A-2.1 Weigh accurately a sample containing about 2·5 g of phorate into a 250-ml volumetric flask. Dilute to the mark with acetone and mix.

A-2.1.1 Place 150 ml water, 5 drops of concentrated nitric acid and exactly 50 ml of standard silver nitrate solution into a 500-ml Erlenmeyer flask. Immerse in a water-bath maintained at 50°C until a constant temperature is attained. Pipette 50 ml of diluted sample (**A-2.1**) into the Erlenmeyer flask. Swirl the flask while adding solution. Stopper and allow to remain in the 50°C water-bath for 15 minutes. Add 5 ml nitrobenzene, stopper tightly and shake vigorously for about 1 minute. Add 1 ml of ferric nitrate solution, shake for 1 minute and begin titrating with standard

ammonium thiocyanate solution. As the end point is approached, the solution will assume a light salmon-pink colour. Stopper and shake vigorously, the salmon-pink colour will disappear. Add more standard ammonium thiocyanate solution and shake again. Repeat this process until the colour is not completely discharged on shaking, but avoid adding of excess standard ammonium thiocyanate solution. When a little colour remains, remove this by the addition of a drop or two of standard silver nitrate solution. At this point, one additional drop of standard ammonium thiocyanate solution should reproduce the faint salmon-pink colour, this indicates the end point.

A-2.2 Determination of Impurities — Weigh accurately a quantity of sample containing 0·5 g of phorate into a 250-ml separatory funnel containing 150 ml of benzene. Extract the benzene solution three times by shaking each time with 50 ml of standard potassium hydroxide solution in 10 percent potassium nitrate, then once with 50 ml of water. Collect the aqueous layers in a 500-ml glass-stoppered Erlenmeyer flask. Neutralize the combined aqueous layers with nitric acid, using congo red paper. Add 5 drops of acid in excess, then add exactly 5 ml of standard silver nitrate solution. Add 5 ml of nitrobenzene and shake vigorously for about 1 minute, and continue titration as in **A-2.1**.

A-2.3 Calculation

A-2.3.1 Phorate content, percent by mass

$$= \left[\frac{6{\cdot}510 \times (V_1F_1) - (V_2F_2)}{M_1}\right] - \left[\frac{1{\cdot}302 \times (v_1F_1) - (v_2F_2)}{M_2}\right]$$

where

V_1 = volume in ml of standard silver nitrate solution consumed,

F_1 = factor of normality of standard silver nitrate solution,

V_2 = volume in ml of standard ammonium thiosulphate solution consumed,

F_2 = factor of normality of standard ammonium thiocyanate solution,

M_1 = mass in g of the material taken for the test (**A-2.1**),

v_1 = volume in ml of standard silver nitrate solution consumed for determination of impurities (**A-2.2**),

v_2 = volume in ml of standard ammonium thiocyanate solution consumed for determination of impurities (**A-2.2**), and

M_2 = mass in g of the material taken for determination of impurities (**A-2.2**).

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